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Diaminoanthraquinone Derivatives as Components of Colored Alkyd Resin

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INVENTION SPECIFICATION

PERTAINING TO A CERTIFICATE OF AUTHORSHIP

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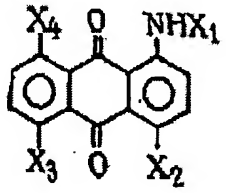
(53) 547.673.5(088.8)

(56) Kogan, M. I. Chemistry of Dyes, Moscow, 1956, pp. 515-516.

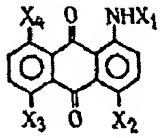
(54) [Title of the Invention]
Diaminoanthraquinone Derivatives as Components of Colored Alkyd Resin

(57) Diaminoanthraquinone derivatives of the general formula



where X_1 = COOH=CHCOOH when
X_2 = NHCOCH=CHCOOH and X_3 = X_4 = H or
X_2 = X_4 = H and X_3 = NHCOCH=CHCOOH;
X_1 = CH_2CH_2OCOCH=CHCOOH when
X_2 = $NHCH_2CH_2OCOCH$=CHCOOH,
X_3 = X_4 = OCOCH=CHCOOH, as components of colored alkyd resins.

The invention relates to diaminoanthraquinone derivatives of the general formula:



(1)

where X_1 = COOH=CHCOOH when X_2 = NHCOCH=CHCOOH and X_3 = X_4 = H (Ia) or X_2 = X_4 = H and X_3 = NHCOCH=CHCOOH (Ib); X_1 = CH_2CH_2OCOCH=CHCOOH when X_2 = $NHCH_2CH_2OCOCH$=CHCOOH, X_3 = X_4 = OCOCH=CHCOOH (Ic), which are components of colored alkyd resins.

The objective of the invention is to create new components of colored alkyd resins (CAR) that permit preparation of CAR with a high color quality in one stage without conducting additional coloring operations.

Preparation of the compounds of general formula (I) is illustrated by examples 1-3.

Example 1. 180 mL nitrobenzene, 11.76 g maleic anhydride and 14.28 g 1,4-diamino-anthraquinone are charged to a flask. The reaction mass is heated to 174°C during mixing and held for 35 minutes. A compound of formula Ia in 98.5% yield is obtained.

Calculated, %: C 60.96; H 3.23; N 6.46

$C_{22}H_{14}O_6N_2$

Found, %: C 60.6; H 3.01; N 6.79

The IR spectrum corresponds to structural formula Ia.

Example 2. A process using 1,5-diaminoanthraquinone is run as in example 1. Compound Ib is obtained with a 98.7% yield.

Calculated, %: C 60.96; H 3.23; N 6.46

$C_{22}H_{14}O_8N_2$

Found, %: C 60.52; H 2.93; N 6.81

The IR spectrum corresponds to structural formula Ib.

Example 3. 18 mL nitrobenzene, 23.52 g maleic anhydride and 22.8 g 1,4-diethanol-amino-5,8-dioxyanthraquinone are charged to a flask. The reaction mass is heated to 180°C during mixing and held for 30 minutes. The contents of the flask are cooled, the precipitate

filtered off, washed with hot water and dried. A compound of formula Ic is obtained with a 96.5% yield.

Calculated, %: C 54.55; H 3.47; N 3.74

$C_{34}H_{26}O_{18}N_2$

Found, %: C 54.23; H 3.2; N 4.14

The IR spectrum corresponds to structural formula Ic.

Examples 4 to 12 illustrate preparation of colored alkyd resins using compounds of general formula (I).

The properties of the obtained alkyd resin film are shown in the table, which also shows the properties of the films obtained in similar fashion, containing known dyes of the aminoanthraquinone series.

Example 4. 60 g sunflower oil and 14.62 g pentaerythritol are charged to a flask. The temperature is gradually raised to 150-160°C and 0.06 g of a calcined medium [sic][TN] is charged, whereupon the temperature is raised to 245-250°C and the reaction mass held in a stream of inert gas, whereupon it is cooled to 180°C and 25.38 g phthalic anhydride is charged. The temperature is then raised to 240-245°C and held to completion of the polycondensation process. After this, the reaction mass is cooled to 210°C, 0.2 g of compound Ib is added and held until an acid number of no more than 20 mg KOH per g of resin is reached. The temperature is then reduced to 140°C and xylene charged. The mass is mixed for 1 hour until a homogeneous solution is obtained. The obtained structurally colored coating of the type PF-060 is investigated in the form of a film applied to a glass plate.

The test results are shown in the table.

In examples 5-12 and in the comparative examples 13-15 the process was run similarly.

[TN] Translator's Note: **кальцинированная с да** (calcined soda, i.e. anhydrous sodium carbonate) might have been intended here, rather than **кальцинированная среда** (calcined medium, whatever that might mean)

Example	Dye	Addition of dye, g/100 g	Hardness of film according to a pendulum instrument
1	2	3	4
4	1b	0.2	0.25
5	1b	0.7	0.26
6	1b	1.0	0.27
7	1c	0.2	0.24
8	1c	0.7	0.27
9	1c	1.0	0.3
10	1a	0.2	0.26
11	1a	0.7	0.27
12	1a	1.0	0.25
13	1,5-diaminoanthraquinone	0.7	0.20
14	1,4-diaminoanthraquinone	0.7	0.23
15	1,4-diethanolamino-5,8-dioxyanthraquinone	0.7	0.22

Note: The external appearance of the coating: according to examples 4-12 transparent, colored without mechanical impurities, according to examples 13-15 – brown, nontransparent with mechanical impurities.

It is apparent from the table that use of the known dyes of the aminoanthraquinone series does not permit preparation of high-quality colored films. The advantage of the coatings containing the proposed compounds is higher mechanical strength of the films.